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Fracture Toughness Estimation From Ceramic Tensile Specimens by Fractographic Analysis

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ARL-TR-1405 June 1997

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ARL-TR-1405 **June 1997**

Fracture Toughness Estimation From Ceramic Tensile Specimens by Fractographic Analysis

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Abstract

Empirical criteria are presented for selection of uniaxial tensile failure origins that will yield valid fracture toughness estimations. Using these criteria, estimations of fracture toughness based on fractography of an AIN, two SiCs, a AIN-SiC solid solution and a particulate composite, and a TiB_2 were performed. Comparison of the fracture toughness values estimated from fractography with fracture toughness measured by the single-edge precracked beam (SEPB) method will be shown. Close agreement is observed between estimates made from fractographic analysis and measurements made on specimens with large artificially induced flaws if the fracture toughness is 4 MPa$\sqrt{m}$ or below. For materials in which the SEPB fracture toughness is greater than about 4.8 MPa$\sqrt{m}$, estimates by fractographic analysis significantly underestimate the SEPB fracture toughness. In attempting to rationalize this discrepancy, the importance of rigorously applying the empirical criteria was reemphasized.

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1. Introduction

Fractography is widely used to identify failure initiating flaws and flaw populations in advanced ceramics. However, fractography is seldom used to determine the fracture toughness of ceramics. This is surprising, since fractography has long been used to estimate the fracture energy of brittle materials, glasses in particular. One such study of the use of fractography to calculate fracture surface energy for a wide variety of ceramics and glasses was conducted by Mecholsky et al. [1]. They were able to show a good correlation between the size of the outer fracture mirror in bend specimens and K_{IC} as measured in a double cantilever beam (DCB) test. Rice [2] presented a comprehensive paper on the fractographic determination of K_{IC} in ceramics at the Second Conference on the Fractography of Glass and Ceramics in 1990. He showed that, for bend bars with large flaws, good agreement was obtained between K_{IC} estimated from fractographic analysis and DCB tests. However, as the flaw sizes decreased, there was a marked tendency for an increasing disparity between the fracture toughness or fracture energies estimated by fractographic analysis and DCB measurements.

In the past several years, two very useful documents [3, 4] have been prepared by researchers at the U.S. Army Research Laboratory (ARL) and the National Institute of Science and Technology, which provide handbook-type guidance in the use of fractography and the relationship between fractography and fracture toughness, but they provide no specific data correlating K_{CI} and fracture origins. Swab and Quinn [4] contains a very useful appendix, which discusses, in detail, various circumstances that might cause discrepancies in estimates of fracture origin size predictions from fracture mechanics, and fractographically observed flaw sizes. Virtually all examples of the use of fracture mechanics to estimate critical flaw sizes or fracture energies/toughness discussed in the previously listed references involved bend tests where fracture origins were often associated with surface machining damage.

With the growing use of tensile testing in the determination of strength statistics for advanced ceramics, a large number of samples of known uniaxial tensile stress and unambiguous failure

origins have become available for analysis. Little work has been reported on using fractography of uniaxial tensile test specimens to estimate fracture toughness. One early work that used fractography of carefully lapped uniaxial tensile specimens of HS130* Si_3N_4 (an early version of NC132* material) [5] yielded an estimated K_{IC} of 2.8 MPa$\sqrt{m}$. No correlation with measured values was made. Recent work by Katz and his coworkers [6] compared K_{IC} estimated by fractographic analysis and those measured by a single-edge precracked beam test (SEPB) for an AIN, a 50%/50% AIN-SiC solid solution and a 25% SiC particulate toughened AIN composite. For all three materials, the agreement between estimated and measured K_{IC} values was within 10%. In a subsequent study of two SiC materials, it was observed that the SiC with a K_{IC} of 4.9 MPa$\sqrt{m}$ showed the largest deviation (−12%) from measured SEPB values of any material that we had tested [7]. A study by Oishi et al. [8], conducted on Y-TZP material, also compared measured K_{IC} (by SEPB) with estimates from fractography. Fractographic estimates were as much as 35% less than the SEPB values.

The purpose of the work discussed in this report was to extend the range of polycrystalline ceramics for which estimates of K_{IC} could be compared to measured values. TiB_2, which has a moderately high K_{IC} and a moderate tensile strength, was chosen as a material that would likely produce a reasonable percentage of internal, as opposed to machining induced, failure origins. Discrepancies between estimated and measured values for TiB_2 and other "high" K_{IC} ceramics are rationalized in terms of subcritical crack growth and/or R-curve behavior. Consideration of the consequences of subcritical crack growth reemphasizes the difference between the failure initiating flaw and the critical flaw, even though the later contains the former. Fractographs showing a hierarchy of concentric flaws in a TiB_2 specimen will be presented to emphasize this point.

2. Experimental Technique

The tensile specimens used in this work were right circular cylinders, measuring between 100 and 120 mm in length and 8.8–9.2 mm in diameter. 40 mm on each end of the test specimen were

* Norton Co., Worcester, MA 01606.

inserted into a steel piston and adhesively bonded in place with high-strength epoxy* [9–11]. The adhesively bonded specimen-piston assembly is inserted into the pressure chamber of an ASCERA Hydraulic Tensile Tester.† Pressure is applied via a hydraulic fluid and increased at a rate of approximately 3 MPa per second until the specimen is broken apart by the hydraulic pressure acting against the piston faces. Figure 1 shows a schematic of the pressure chamber with the adhesively bonded specimen-piston assembly in place. The tensile fracture stress is calculated using the pressure at the instant of failure and the geometric parameters of the specimen-piston assembly [9–11]. Although this tensile testing method is inherently self-aligning, a small amount of eccentricity may occur, arising from either the specimen being slightly curved or the epoxy bonding being slightly off-center in the pistons. Procedures to correct for this source of error exist and have been described by several investigators [9–12]. Where required, such corrections have been applied to all tensile data used in estimating fracture toughness discussed in this report. Typically, these corrections are in the 3–5% range and in no case exceeded 10%. The stress is related to the pressure and test geometry by the following equation:

$$\sigma = \sigma_{NOM} + \Delta\sigma = (A - A_s)/A_s \cdot P + \Delta\sigma, \quad (1)$$

where σ_{NOM} is the nominal fracture stress, $\Delta\sigma$ is the correction for eccentricity which is equal to $\sigma_{NOM} \times f$(fracture origin location, specimen eccentricity and diameter), A is the cross-sectional area of the piston, A_s is the cross-sectional area of the specimen, and P is the pressure at failure, respectively.

SEPB tests [13] were performed to measure the fracture toughness of materials from the same lots of materials used for tensile testing. At least six valid [14] SEPB tests and more usually 10–14 valid tests were performed for each material using 3- × 4- × 25-mm beams. Precracking was accomplished by indenting the 3-mm face of each specimen with a Vickers diamond indenter (with the diagonals parallel to the edges of the beam) and loaded in a double anvil configuration to

* ARALDITE AV 118, Ciba Geigy Corp., Madison Heights, MI 48071.

† ASCERA Hydraulic Tensile Tester, Robertsfors, Sweden.

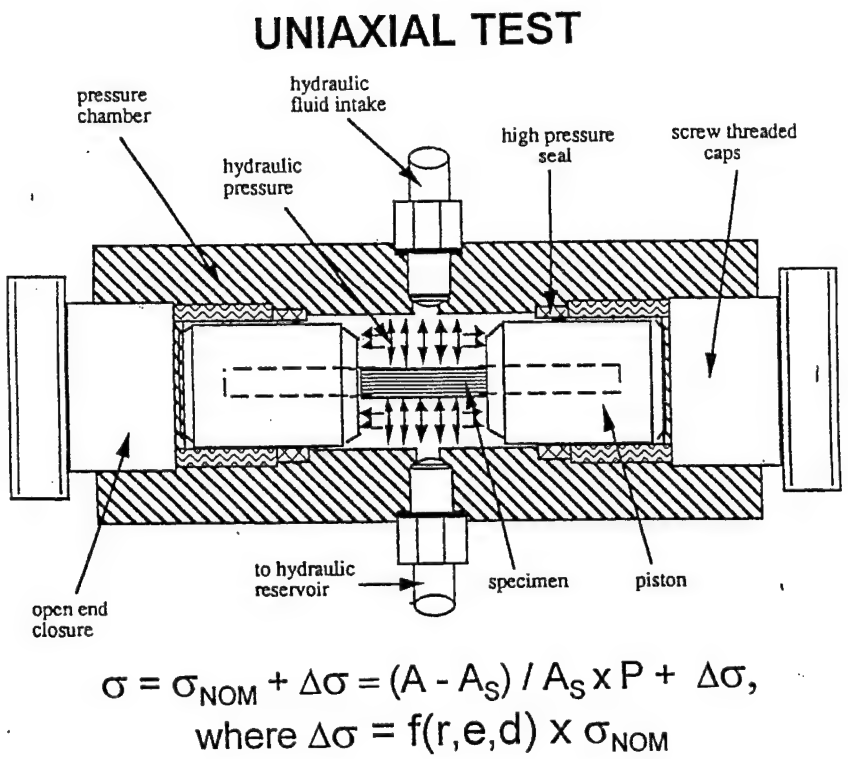

Figure 1. Hydraulic Pressure Chamber With Specimen-Piston Assembly.

"pop-in" a straight crack [15]. "Pop-in" and fracture toughness measurements were carried out on Instron model 8502 and 4201 testing machines,* respectively. Detailed descriptions of the SEPB test procedure are available in Bar-On et al. and Quinn et al. [15, 16].

Fractographic examination of the fracture surfaces of the tensile bars was carried out in a JEOL 840A SEM.† Fracture surfaces were cleaned with acetone and coated with Au-Pd or carbon prior to SEM examination. Fractographic analysis of the tensile failures are conducted prior to carrying out K_{IC} measurements by SEPB. This avoids any subconscious bias in the measurement of the critical flaw size for the K_{IC} estimate.

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* Instrom Corp., Canton, MA 02021.

† JEOL USA, Inc., Peabody, MA 01961.

The materials data and their source used in this work are listed in Table 1. The methodology used to estimate fracture toughness from the fractography of failed uniaxial tensile bars is presented in the following section.

Table 1. Materials and Property Data Used in This Study

Material	Source	$T-\sigma_{CHAR}$	m	AGS[a] (μm)	Reference
HP TiB_2	Cercom	275	14.9	15	This study
HP AlN	Dow	234	12.7	1.9	Katz et al.
HP 50AlN/50SiC	Dow	361	26.4	0.5	Katz et al.
HP 75AlN/25SiC	Dow	371	9.9	2[b] and 5[c]	Katz et al.
HP SiC Pad B	Cercom	304[d]	9.6[d]	4.0	Cho, Katz, and Bar-On
HP SiC Pad N	Cercom	347[d]	9.6[d]	4.0	Cho, Katz, and Bar-On

Note: HP = hot pressed; $T-\sigma_{CHAR}$ = tensile characteristic strength; m = Weibull Modulus; and AGS = average grain size.
[a] Based on the company product literature.
[b] AGS of AlN.
[c] AGS of SiC.
[d] Combined test data obtained from specimens machined by two vendors.

3. Criteria for Estimation of K_{IC} by Fractographic Analysis

Only a fraction of the fracture surfaces resulting from uniaxial tensile failures are suitable for estimating K_{IC}. Not all samples have an obvious failure origin; this is often the case for samples failing from surface "defects." In a previous report [6], the authors and their coworkers defined a set of empirical conditions, which restrict those samples to be used for K_{IC} estimation. Later, this report will show that, in fact, these restrictions are necessary for close estimation of K_{IC} (within approximately 10%), and if they are relaxed, the difference between the estimated values and the values measured by SEPB tests increases significantly. The criteria for selecting samples for fracture toughness estimates are:

(1) The failure must be from an internal flaw.

(2) Fracture origins must exhibit fully developed "mirror," mist, and hackle regions.

(3) The mirror has to be "essentially" circular (samples with ellipsoidal mirrors are occasionally encountered and are not used).

(4) The mirror, mist, and hackle regions must be close to normal to the specimen axis. (This ensures that the onset of unstable fracture occurred under a pure uniaxial stress field.)

(5) The largest linear dimension of the failure initiating flaw at the center of the mirror is used as the crack length in the K_{IC} calculation.

The toughness, KIC, is calculated using the Sneddon closed-form solution for a penny-shaped crack [17]:

$$K_{IC} = 2\,\sigma_c\,(a_c/\pi)^{1/2}, \tag{2}$$

where σ_c is the failure stress and a_C is one-half the principal flaw dimension. It is worth noting that criteria (1) and (4) address issues raised by Rice with regard to improving the accuracy of fractographic estimation of K_{IC} [2].

Figure 2 shows a specimen of AIN which exhibits a well-defined mirror, mist, and hackle, as well as a circular mirror. This typifies an acceptable specimen for K_{IC} estimation. Figure 3 shows a specimen of an AIN-SiC solid solution that has an elliptical mirror and is therefore unacceptable for use in estimation of K_{IC}. Figure 4 shows a specimen of TiB_2 which has what we refer to as a "pseudo mirror." In this case, there is a circular area which suggests a mirror, but upon close examination, it can be seen that no mirror, mist, and hackle regions are truly present, only mist and hackle. It will be shown later that such fracture origins are associated with significant underestimation of K_{IC}.

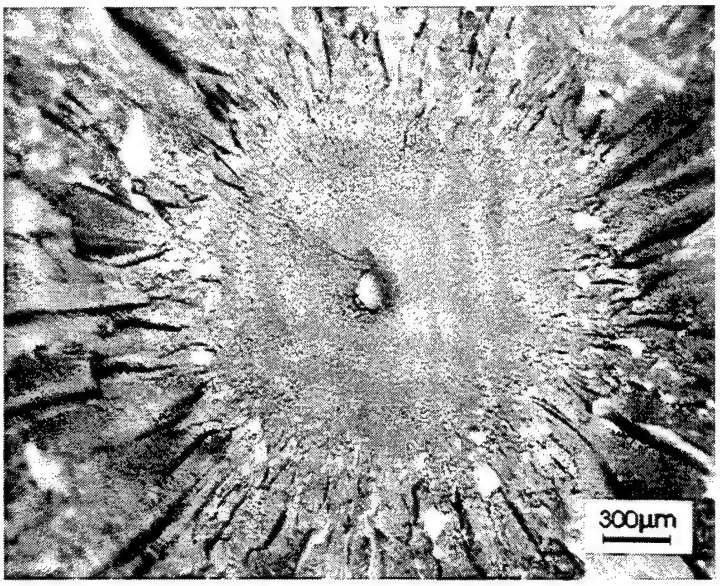

Figure 2. Circular Fracture Mirror in AIN.

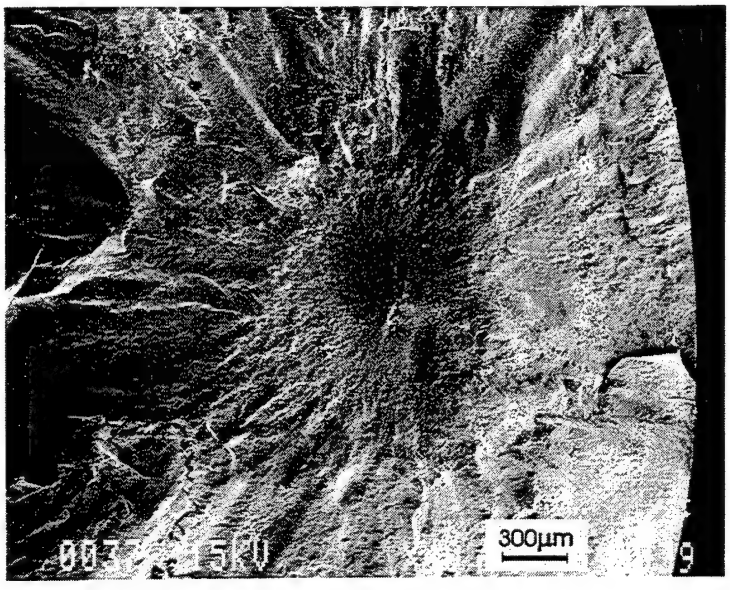

Figure 3. Elliptical Fracture Mirror in AIN-SiC Solid Solution Unacceptable for Analysis.

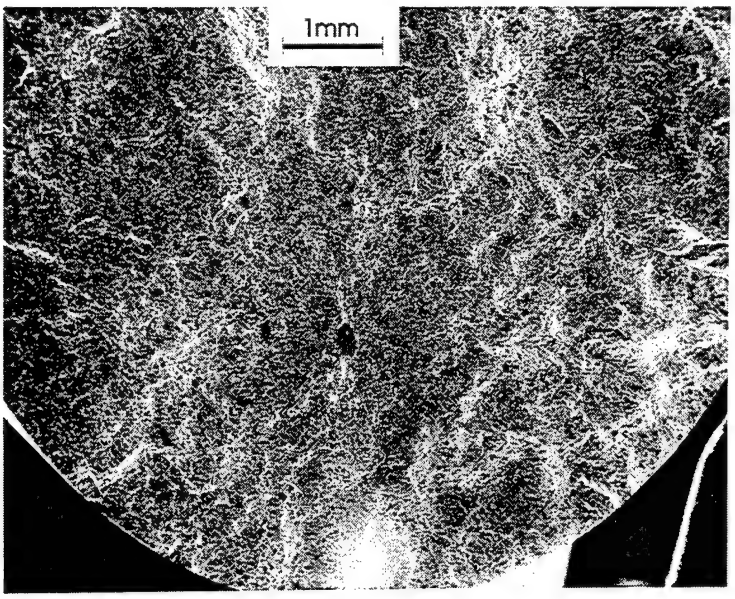

Figure 4. "Pseudo" Mirror in TiB_2 Shows Mist and Hackle But No Mirror.

Whether or not a given material has a mirror associated with the fracture origin can be a function of the chemistry, phase content, and processing history. For example, we have examined the fracture origins in three different SiCs. A Dow experimental SiC* did not exhibit a mirror or mist region. Cercom Pad B† material did exhibit mirror, mist, and hackle (Figure 5), whereas Cercom Pad N† material exhibited no mirror but only mist and hackle (Figure 6).

4. Estimation of K_{IC}

Using the previous criteria, estimates of K_{IC} were made for the six materials listed in Table 1. SEPB K_{IC} measurements were also made on these six materials. Figure 7 presents a plot of the fractographic estimations vs. the measured SEPB values. Very good agreement between the

* Dow Chemical Co., Midland, MI 48674.

† Cercom, Inc., Vista, CA 92083.

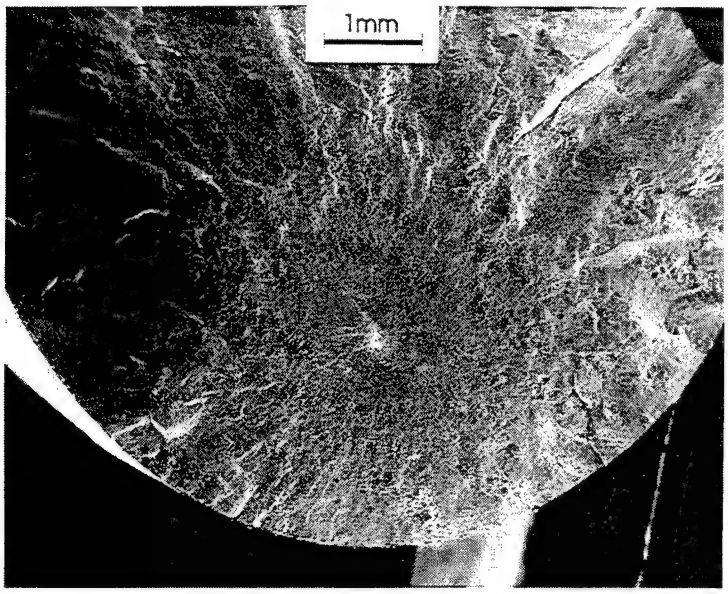

Figure 5. SiC Pad B Shows Mirror, Mist, and Hackle.

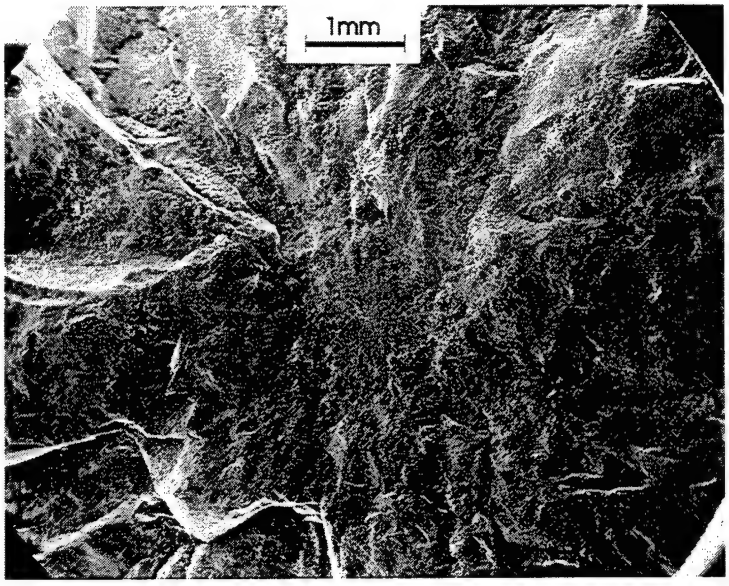

Figure 6. SiC Pad N Shows Only Mist and Hackle.

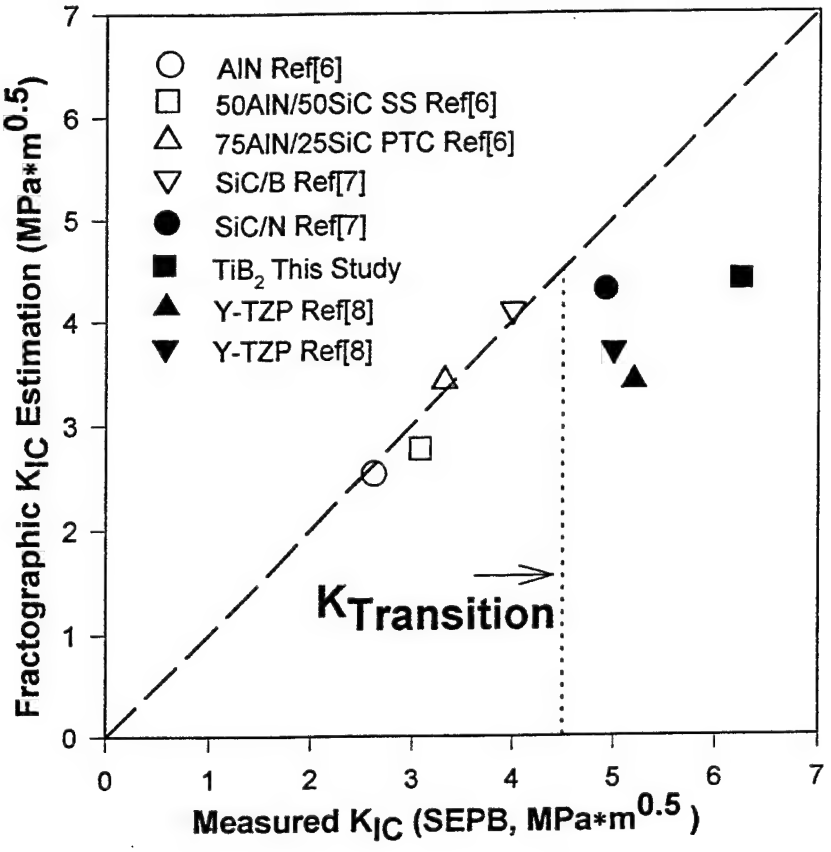

Figure 7. Fracture toughness comparison between fractographic estimates and SEPB measurements.

estimated and measured values is obtained for K_{IC} values determined by SEPB of less than ~4.5 MPa$\sqrt{m}$. Around this value, the estimates of K_{IC} by fractography begin to substantially underestimate the measured values.

This behavior led us to reexamine the fracture surfaces and calculations that we had used in estimating K_{IC}. Upon reexamination, it became apparent that we had "relaxed" criterion (2) and used specimens exhibiting "pseudo mirrors." This clearly emphasized the importance of rigorously applying all of the empirical criteria listed previously, in estimating K_{IC} using fractography. Rationalizing the observed behavior will be the concern of the balance of the report.

5. Potential Sources of Discrepancy Between Estimated and Measured K_{IC} at "High" K_{IC}

While the tensile test technique of Oishi et al. [8] differed substantially from those used by the present authors and their colleagues, the variation that they found in estimated and measured K_{IC} is similar to that observed by other investigators as one goes from "small" flaws to "large" flaws in Y-TZP. The increase in fracture toughness as the flaw size increases has been related to "micro R-curve behavior" [4]. Rice's data on estimation of K_{IC} as a function of flaw size for TZP and ZTA materials also show evidence of "micro R-curve behavior" [2]. It is reasonable that the difference in fracture toughness encountered between large artificially induced flaws, such as in an SEPB test, and the small naturally occurring flaws used in fractographic estimation would be very sensitive to R-curve behavior. Swab and Quinn [4] point out that one cause of observing critical flaws significantly smaller than those predicted from macroscopic fracture toughness tests is the presence of R-curve behavior. As shown in Table 2, the range of flaws measured on TiB_2 tensile fracture surfaces is significantly smaller than the average flaw anticipated using the average strength and measured fracture toughness. By contrast, in the case of AIN and AIN/SiC solid solution, the observed flaw sizes bracket the predicted flaw size, as anticipated. The presence of behavior consistent with a given mechanism, however, only means that the mechanism is plausible. Other mechanisms may, in fact, be responsible for the observed behavior.

Table 2. Anticipated vs. Observed Flaw Size

	AIN	50 AIN/50 SiC	TiB_2
Average σ_c	230 MPa	360 MPa	280 MPa
Average K_{IC}	2.62 MPa$\sqrt{m}$	3.08 MPa$\sqrt{m}$	6.25 MPa$\sqrt{m}$ calculated
Average a_0	~100 µm	~57 µm	~390 µm
Observed ranges of a_0	42–135 µm	5–200 µm	170–280 µm

Note: σ_c = fracture stress; a_0 = flaw size.

An alternative mechanism may be subcritical crack growth without R-curve behavior. Normally, one associates subcritical crack growth with slow crack growth at high temperatures or in the presence of water or other reactive environment. Neither situation prevailed in the tensile testing reported in this work. All tests were at room temperature and since the fracture origins were well in the volume, not near the surface, no reaction with the hydraulic pressurization fluid was possible. Therefore, the most plausible explanation for the existence of subcritical crack growth, in TiB_2, is R-curve behavior as described by Bennison and Lawn [18]. To settle the issue, an investigation of R-curve behavior in TiB_2 should be undertaken.

6. The Fracture Initiating Flaw vs. the Critical Flaw

In ceramic materials that exhibit subcritical crack growth, whether associated with R-curve behavior or some other cause, the fracture initiating flaw will not be the critical flaw for fracture mechanics purposes. An interesting example of this was encountered in one TiB_2 specimen examined in this study. The fracture origin of the specimen shown in Figure 4 is detailed at increasing magnifications. Figure 8a shows the large grain at the center of the "pseudo-mirror." Figure 8b shows that this grain itself has a true fracture mirror with a small (~20 μm) particle at the center of the mirror. This inclusion showed traces of W, Co, Fe, and Ni (by EDXS), which are all consistent with a fragment of WC grinding media. It is probable that this inclusion was the initiating flaw, but it is much too small to be the critical flaw.

Similarly, the large grain is too small to be the critical flaw predicted by fracture mechanics based on the conventional large flaw toughness measurement. Even the various ellipses that one can imagine seeing, linking large grains surrounding the very large grain in Figure 8a, are not large enough to be the critical flaw. It is evident that care should be taken not to use the terms "fracture initiating flaw" and "critical flaw" interchangeably. They may or may not be equal.

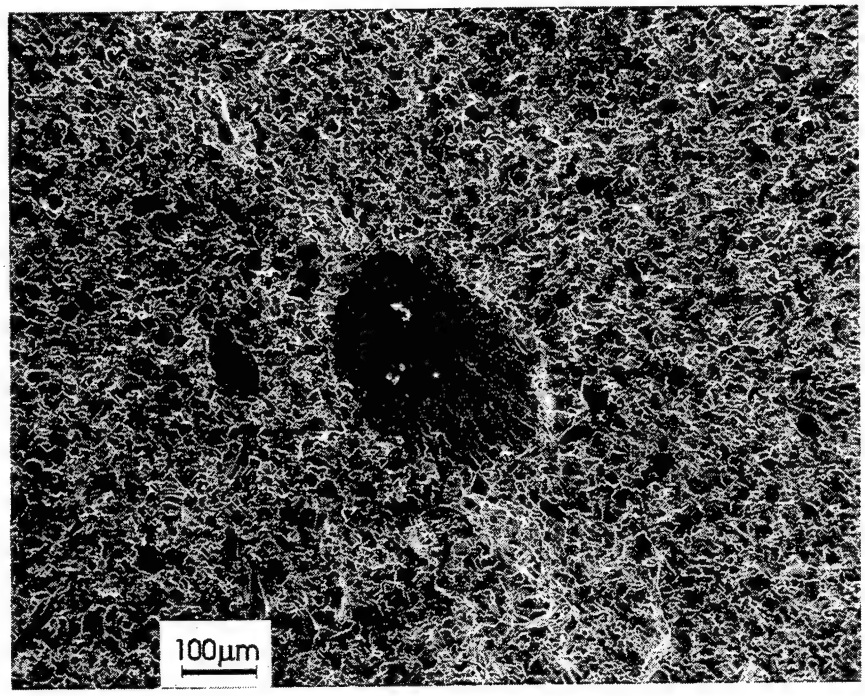

Figure 8a. Large Grain at Origin of Fracture in TiB_2 Specimen.

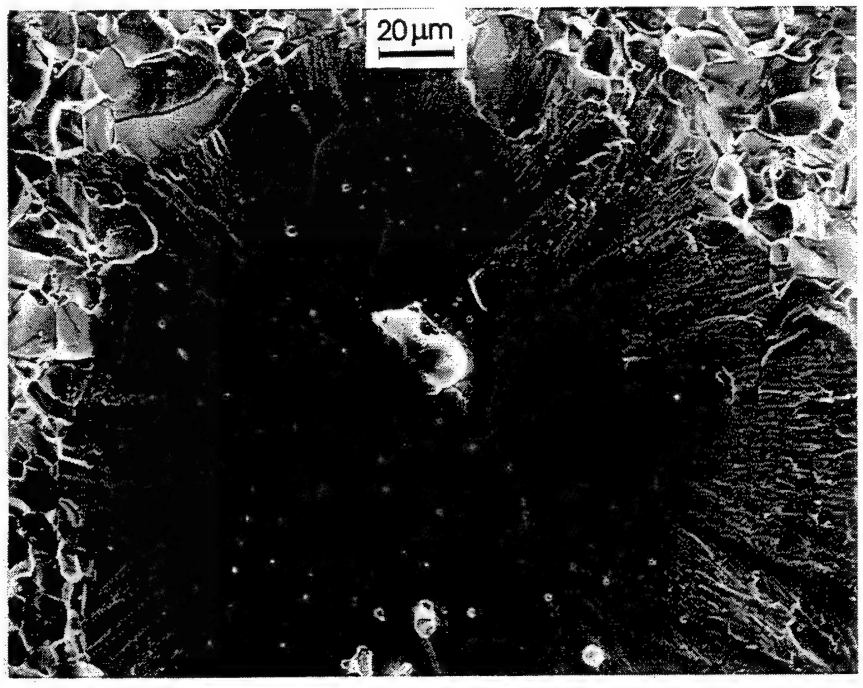

Figure 8b. Particle in Large Grain at Origin of Fracture in TiB_2 Specimen.

7. Summary

We have shown that if the empirical criteria presented in this report are applied, excellent correlation between fractographically estimated and measured K_{IC} can be attained. It is especially critical in applying these criteria to be certain that a true fracture mirror is present. By implication, if the estimated K_{IC} lies on the curve in Figure 7, it can be taken as evidence of a lack of R-curve behavior. Conversely, if the estimated K_{IC} lies substantially below the curve in Figure 7, R-curve behavior may be present. It has also been shown that for "tough" ceramics, the terms "failure initiating" and "critical flaw" are not synonymous.

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REPORT DOCUMENTATION PAGE

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1. AGENCY USE ONLY *(Leave blank)*	2. REPORT DATE	3. REPORT TYPE AND DATES COVERED
	June 1997	Final, Oct 94 - Sep 96

4. TITLE AND SUBTITLE

Fracture Toughness Estimation From Ceramic Tensile Specimens by Fractographic Analysis

5. FUNDING NUMBERS

44LAL
552LBA

6. AUTHOR(S)

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7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

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Aberdeen Proving Ground, MD 21005-5069

8. PERFORMING ORGANIZATION REPORT NUMBER

ARL-TR-1405

9. SPONSORING/MONITORING AGENCY NAMES(S) AND ADDRESS(ES)

10.SPONSORING/MONITORING AGENCY REPORT NUMBER

11. SUPPLEMENTARY NOTES

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13. ABSTRACT *(Maximum 200 words)*

Empirical criteria are presented for selection of uniaxial tensile failure origins that will yield valid fracture toughness estimations. Using these criteria, estimations of fracture toughness based on fractography of an AlN, two SiCs, a AlN-SiC solid solution and a particulate composite, and a TiB_2 were performed. Comparison of the fracture toughness values estimated from fractography with fracture toughness measured by the single-edge precracked beam (SEPB) method will be shown. Close agreement is observed between estimates made from fractographic analysis and measurements made on specimens with large artificially induced flaws if the fracture toughness is 4 MPa$\sqrt{m}$ or below. For materials in which the SEPB fracture toughness is greater than about 4.8 MPa$\sqrt{m}$, estimates by fractographic analysis significantly underestimate the SEPB fracture toughness. In attempting to rationalize this discrepancy, the importance of rigorously applying the empirical criteria was reemphasized.

14. SUBJECT TERMS

fracture toughness estimation, tensile, fractograph

15. NUMBER OF PAGES

25

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
UNCLASSIFIED	UNCLASSIFIED	UNCLASSIFIED	UL

NSN 7540-01-280-5500

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